

Center for Nanophase Materials Sciences: Overview and Needs for Advanced Microscopy

**Linda Horton
Division Director
Center for Nanophase Materials Sciences**

CNMS Microscopy Lead: David Joy

June 23, 2008

CNMS Integrates Nanoscale Science with Synergistic Research

- **Neutron Science**

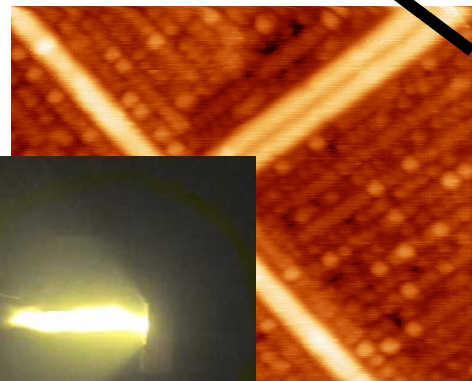
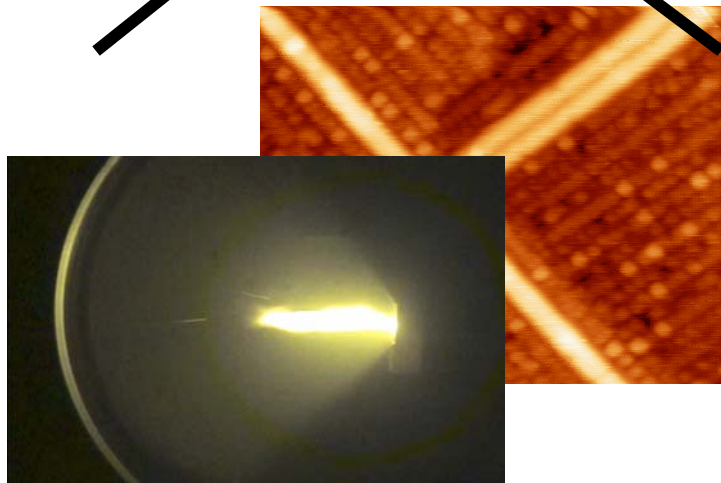
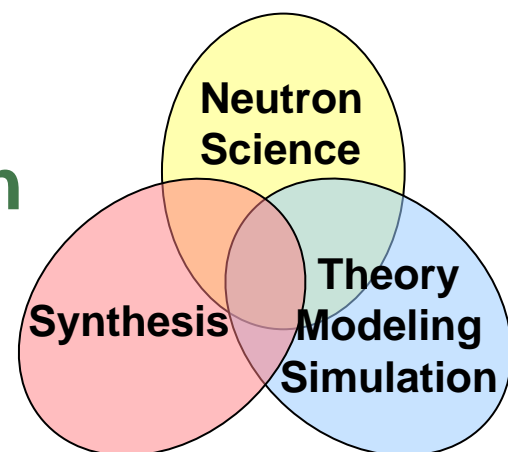
- Opportunity for **world leadership** using **unique capabilities** of neutron scattering
- Complementary to other characterization instrumentation

- **Synthesis Science**

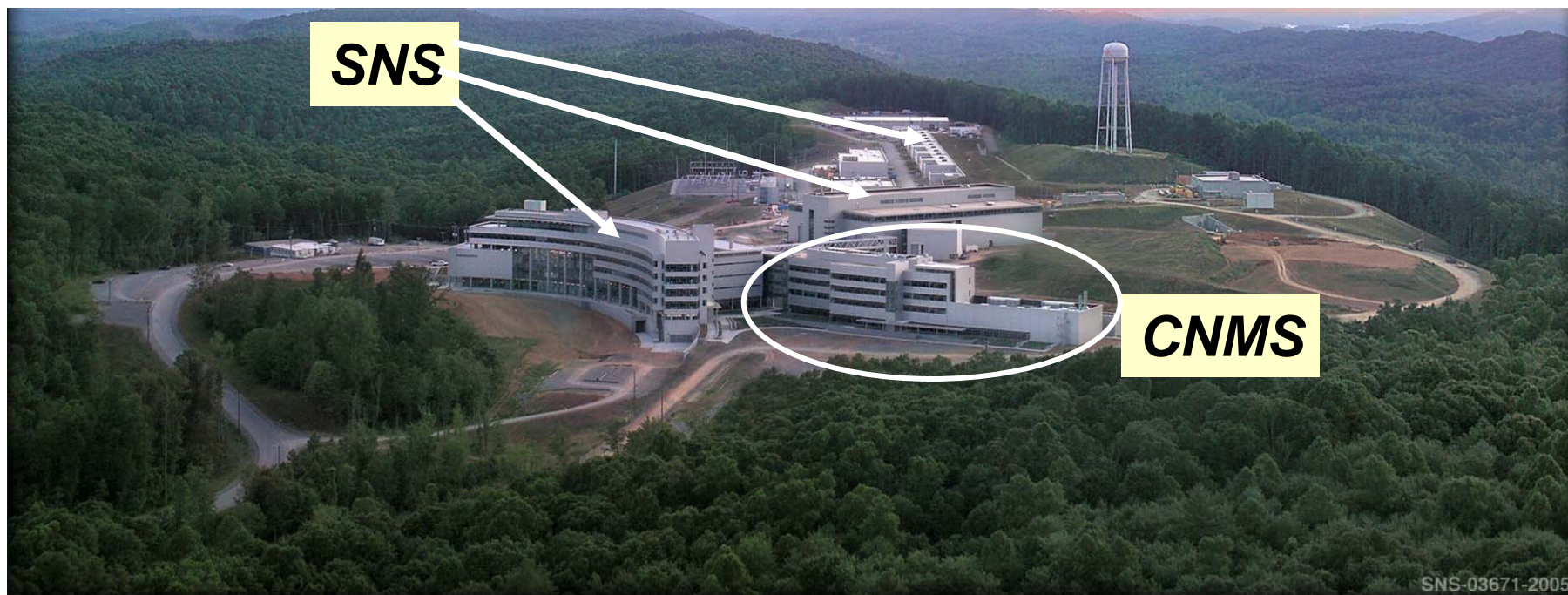
- **Science-driven** synthesis using unique instrumentation and integrated with characterization

- **Theory / Modeling / Simulation**

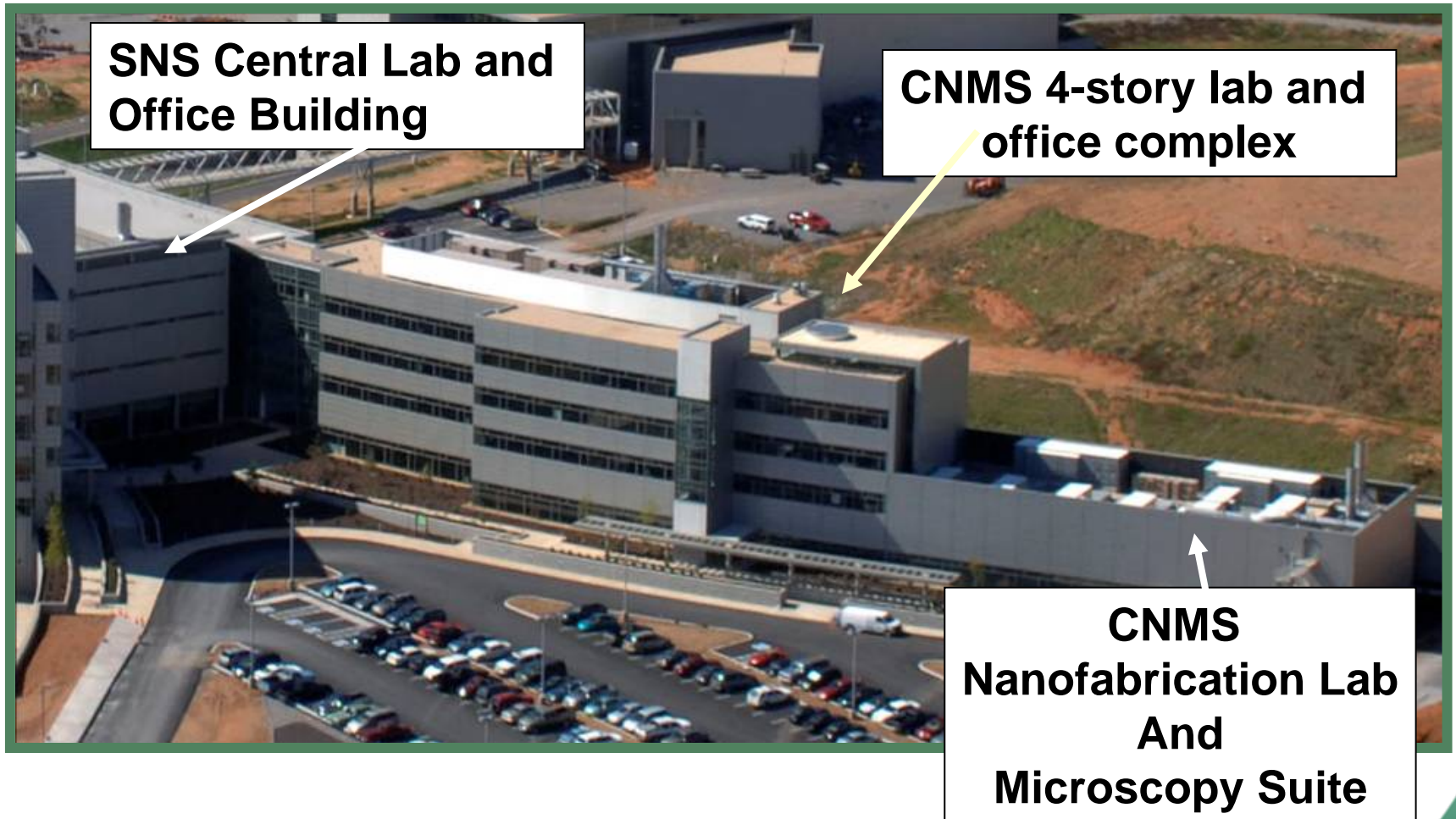
- Stimulate use of theory, modeling and simulation to design new nanomaterials
- Investigate new pathways for materials synthesis



CNMS is located on ORNL's Chestnut Ridge Site with the Spallation Neutron Source



Center for Nanophase Materials Sciences



CNMS Science

- **Understanding, designing, and controlling the dynamics and spatial aspects of functionality in nanoscale systems**
 - **Functional polymeric architectures**
 - **Imaging nanoscale functionality**
 - **Spatial, spectroscopic, dynamics**
 - **Systems as well as individual nanostructures**
 - **Understanding emergent behavior**

Electron Microscopy is Essential to These Goals



CNMS Research Capabilities

Macromolecular Nanomaterials

Synthetic polymeric and bio-inspired materials

Nanomaterials Theory Institute

Theory and Modeling for nanoscience grand challenges

Nanofabrication Research Laboratory and Bio-Inspired Nanomaterials

Controlled synthesis & directed assembly; functional integration of “soft” and “hard” materials

Imaging Nanoscale Functionality

Unique Scanning probe characterization;
in-situ growth of nanostructures

Functional Multiscale Characterization

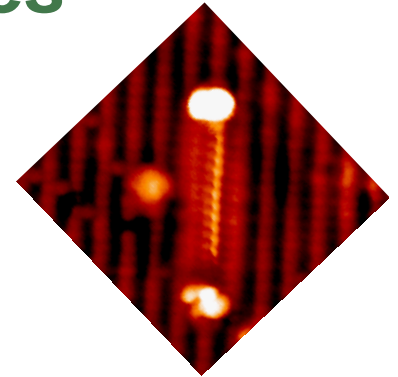
Neutrons, Electron, and X-ray Characterization
Raman, optical characterization
Environmental control, dynamics

Catalytic Nanosystems

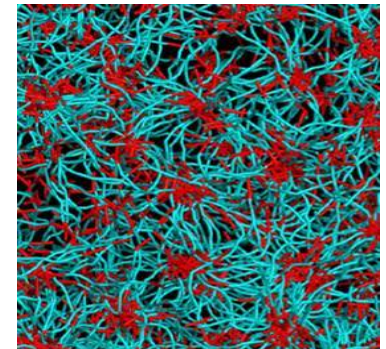
Highly selective catalysts

Functional Hybrid Nanostructures

Nano- tubes, wires, dots, composites; artificial oxide films



STM of Ti adatoms line defect on the surface of TiO_2 ; K. T. Park, M. H. Pan, V. Meunier, and E. W. Plummer, Phys. Rev. Lett. 96, 226105 (2006).



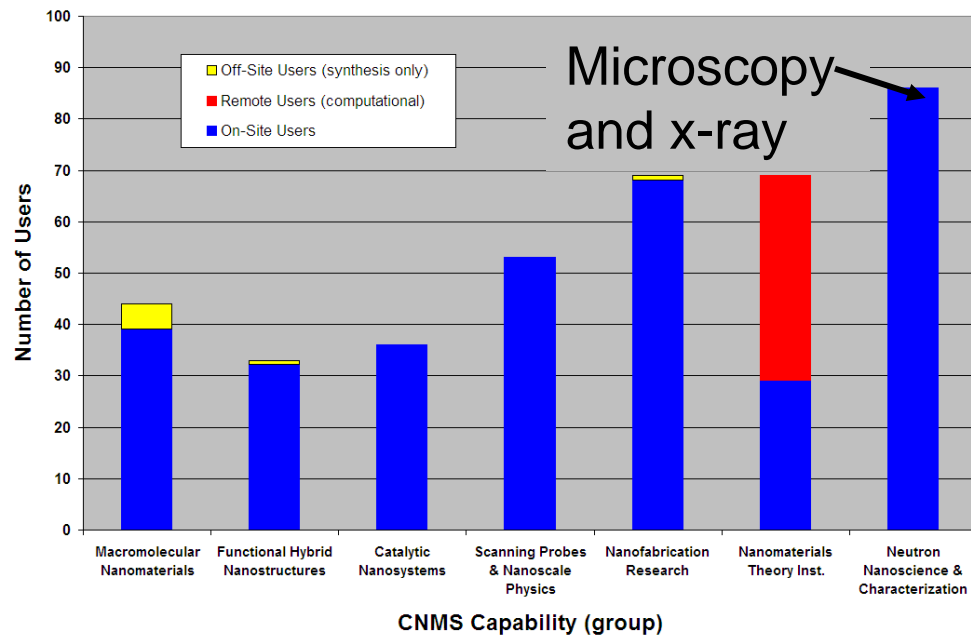
Molecular dynamics simulation of reversible gel formation of triblock copolymers, carried out by CNMS users Lei Guo and Erik Luijten (U. of Illinois-UC)

FY07 CNMS User Statistics: 309 Unique Users (FY08: ~240 thru March)

- ~75% from outside of ORNL; ~75% first time users;
- <70% proposal acceptance

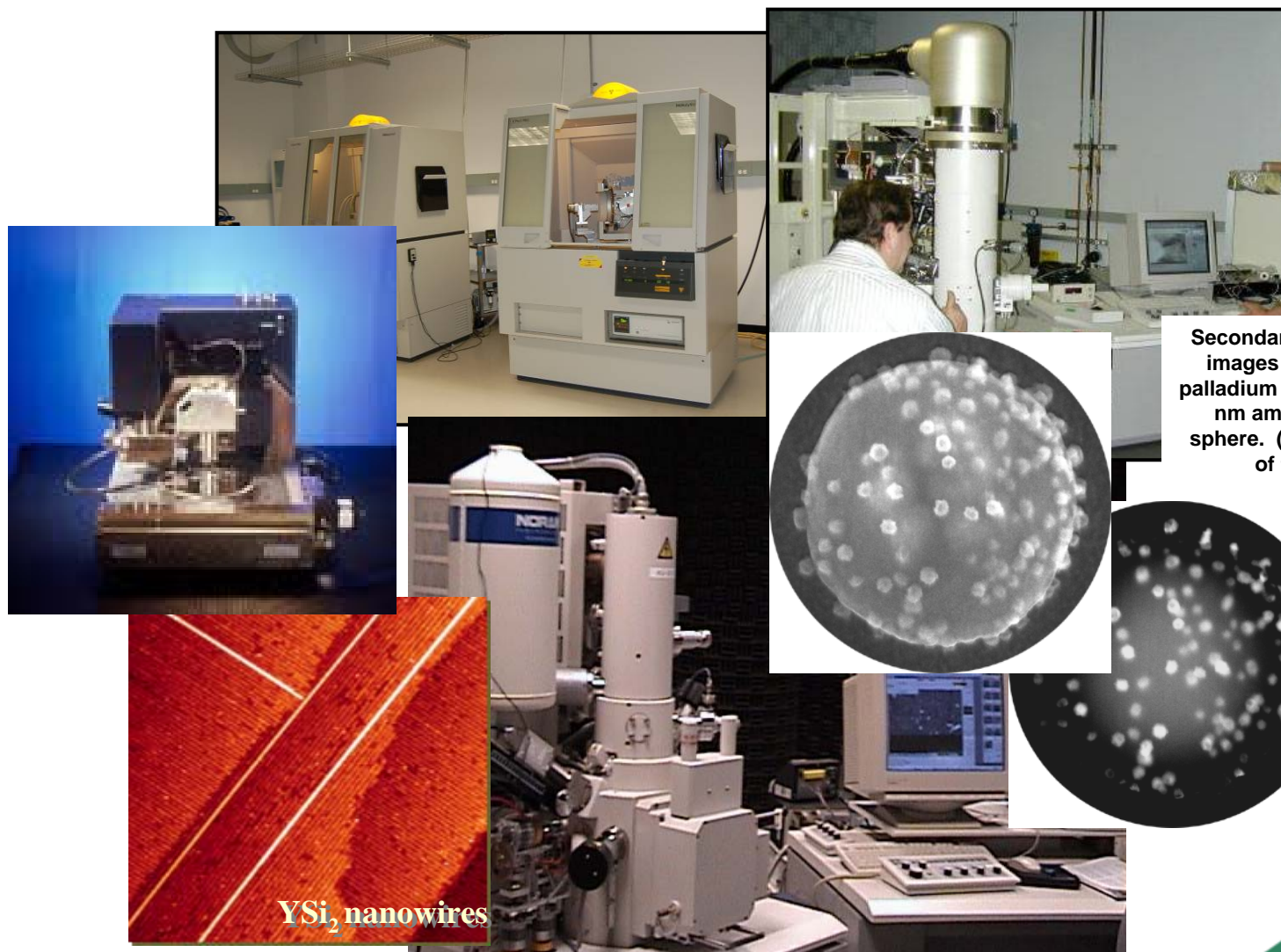
Typical US Geographic
Distribution of User
Proposals

Capabilities Accessed By Users in FY07



Users come from across the
Globe

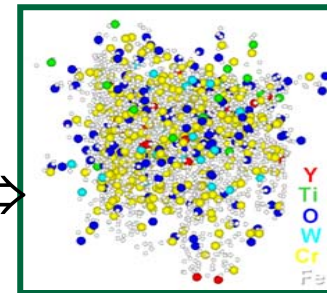
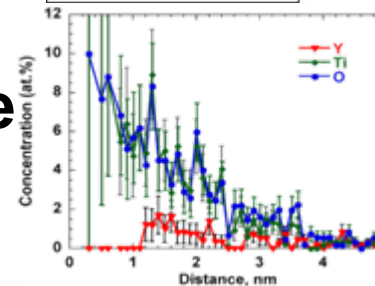
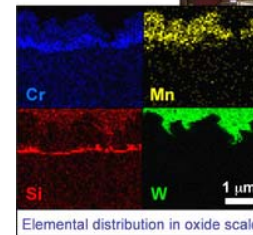
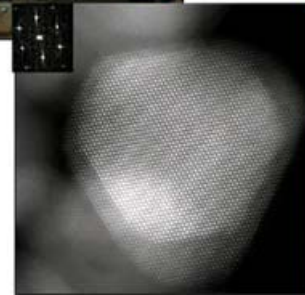
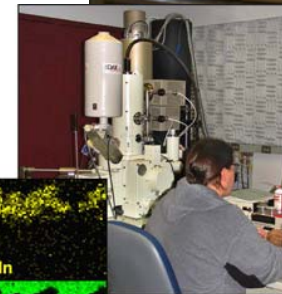
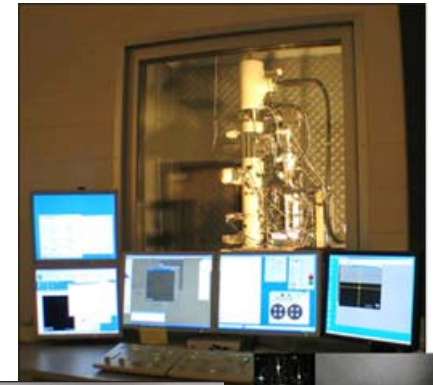
The CNMS Facility Houses Essential Characterization Equipment – including Basic Electron Microscopes



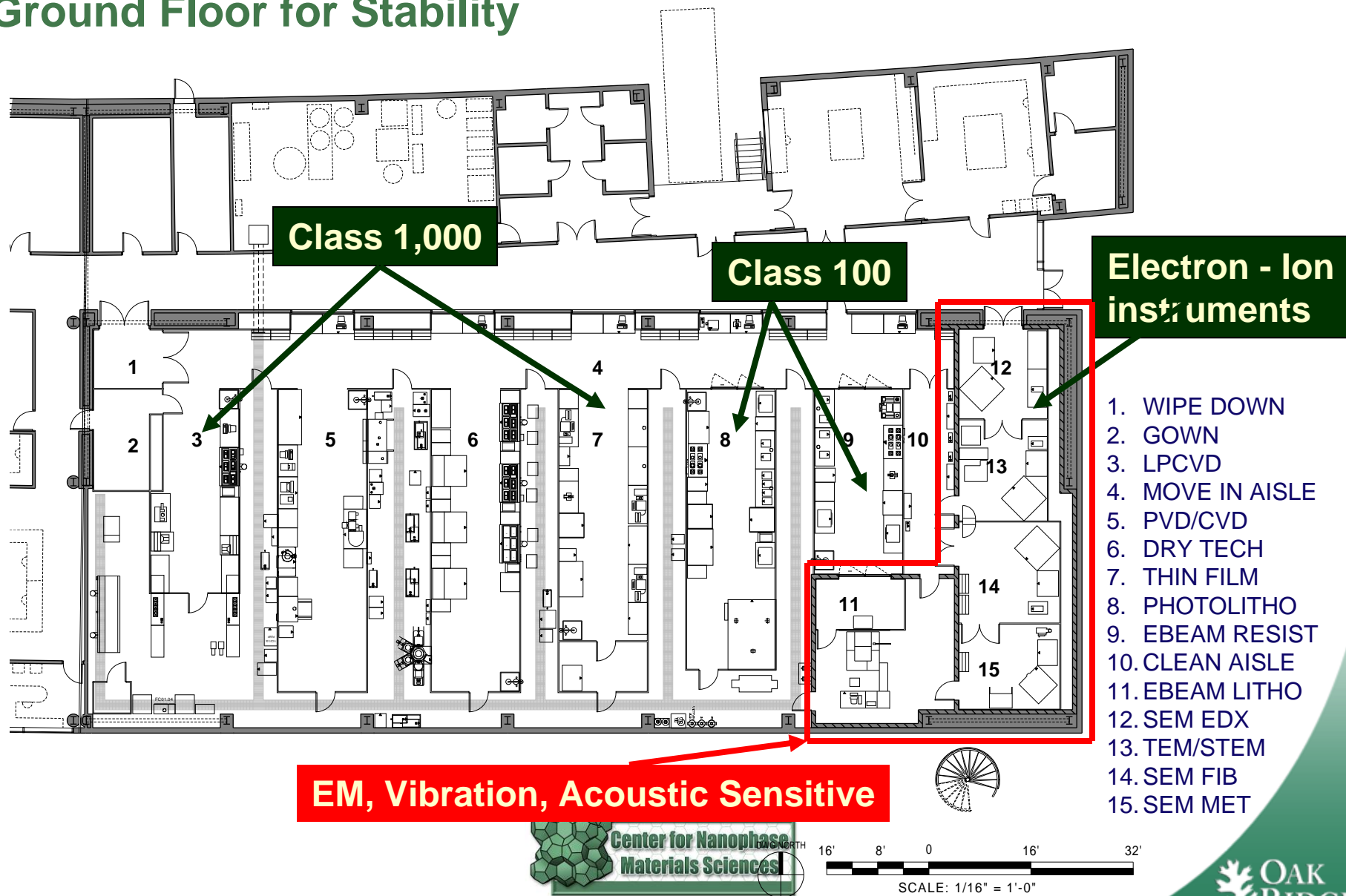
Secondary and z-contrast images of 5 nm hollow palladium particles on a 200 nm amorphous silica sphere. (Images courtesy of the HTML)

CNMS Partners with the Shared Research Equipment User Program for Advanced Electron and Atom Probe Microscopy Capabilities

- **Single User Proposal for both facilities**
- **JEOL 2200FS Aberration-Corrected Scanning Transmission Electron Microscope**
 - 3 addition instruments
 - (200 and 300 keV)
 - Energy-Filtered TEM and EELS
- **JEOL 6500F SEM**
 - 3 additional instruments
- **Imago Local Electron Atom Probe**
- **Dual Beam Focused Ion Beam**



CNMS Clean Room and Electron/Ion Instrumentation: Ground Floor for Stability

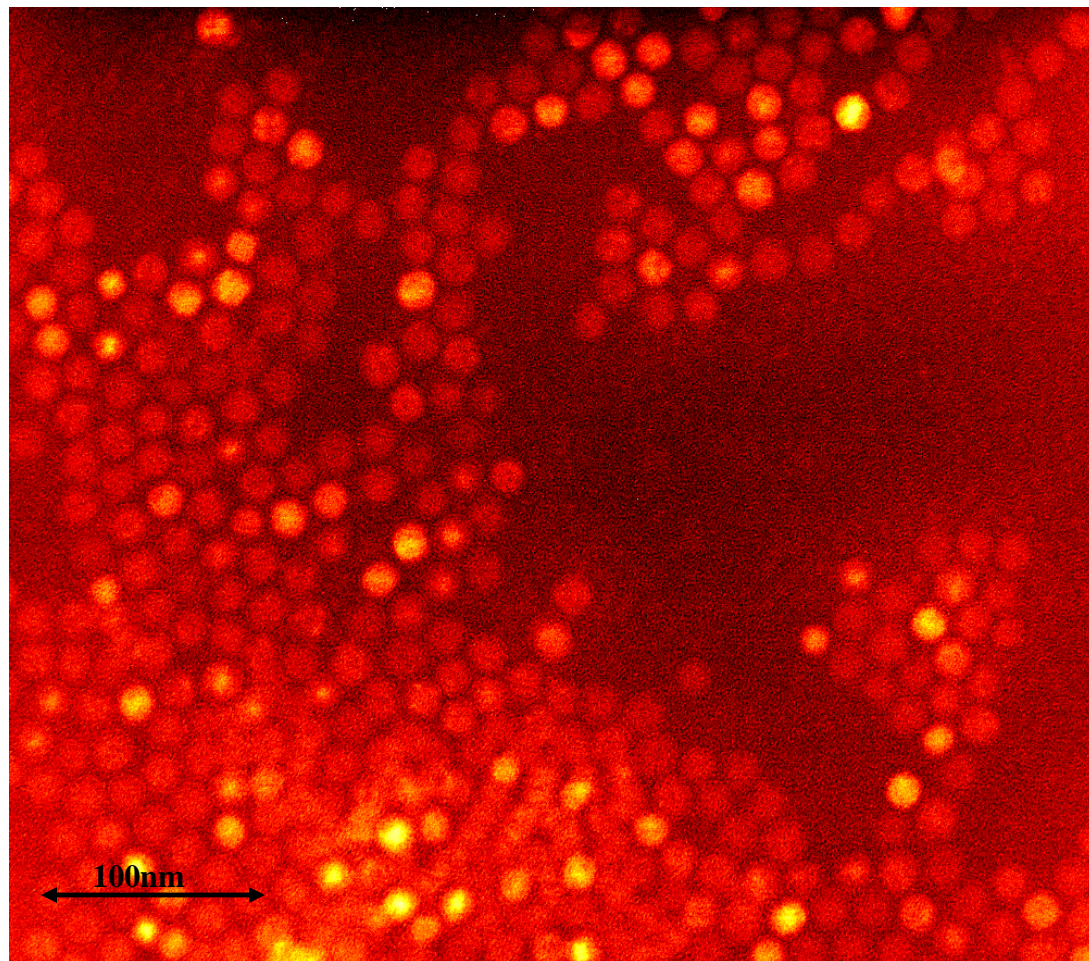


CNMS In-house STEM: HD2000

- 2.3Å bright field lattice resolution
- HAADF “Z contrast” imaging at better than 3Å
- High performance X-ray microanalysis, line profiles, mapping, spectrum imaging

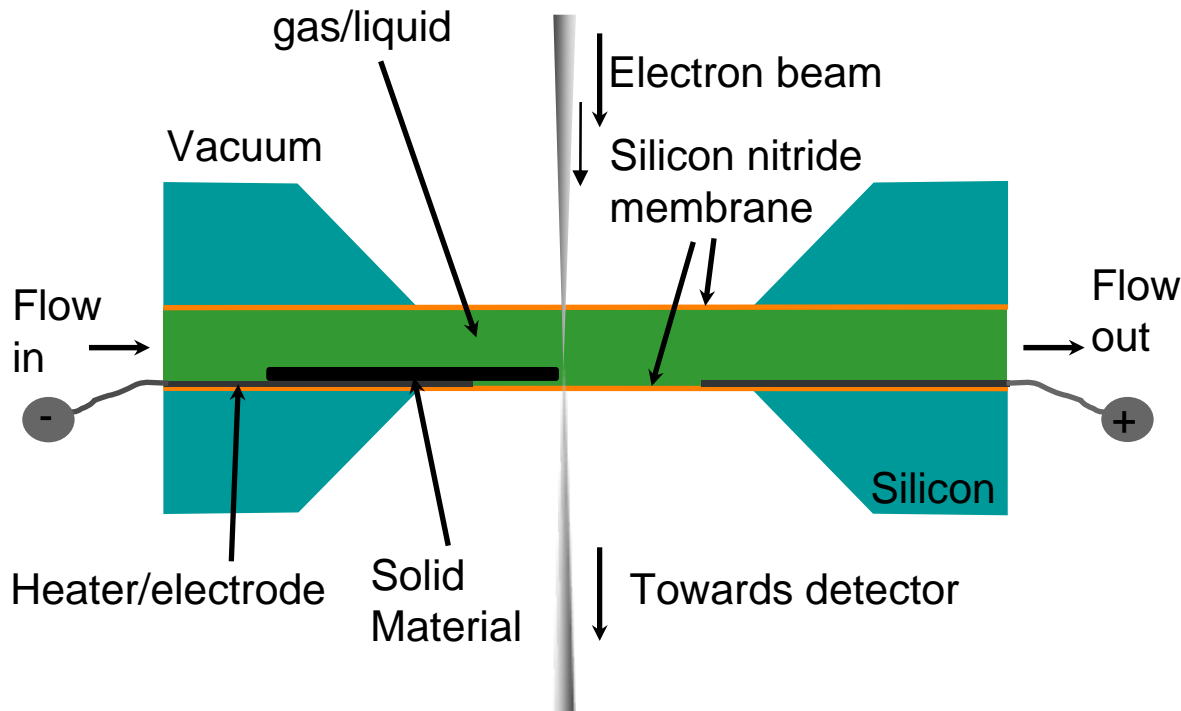
**Z contrast image of mixed Fe_2O_3
and Eu_2O_3 nanospheres**

Courtesy Sameer Mahajan



CNMS In-house STEM

Hitachi HD2000 dedicated FEG STEM, NORAN 6 EDS system



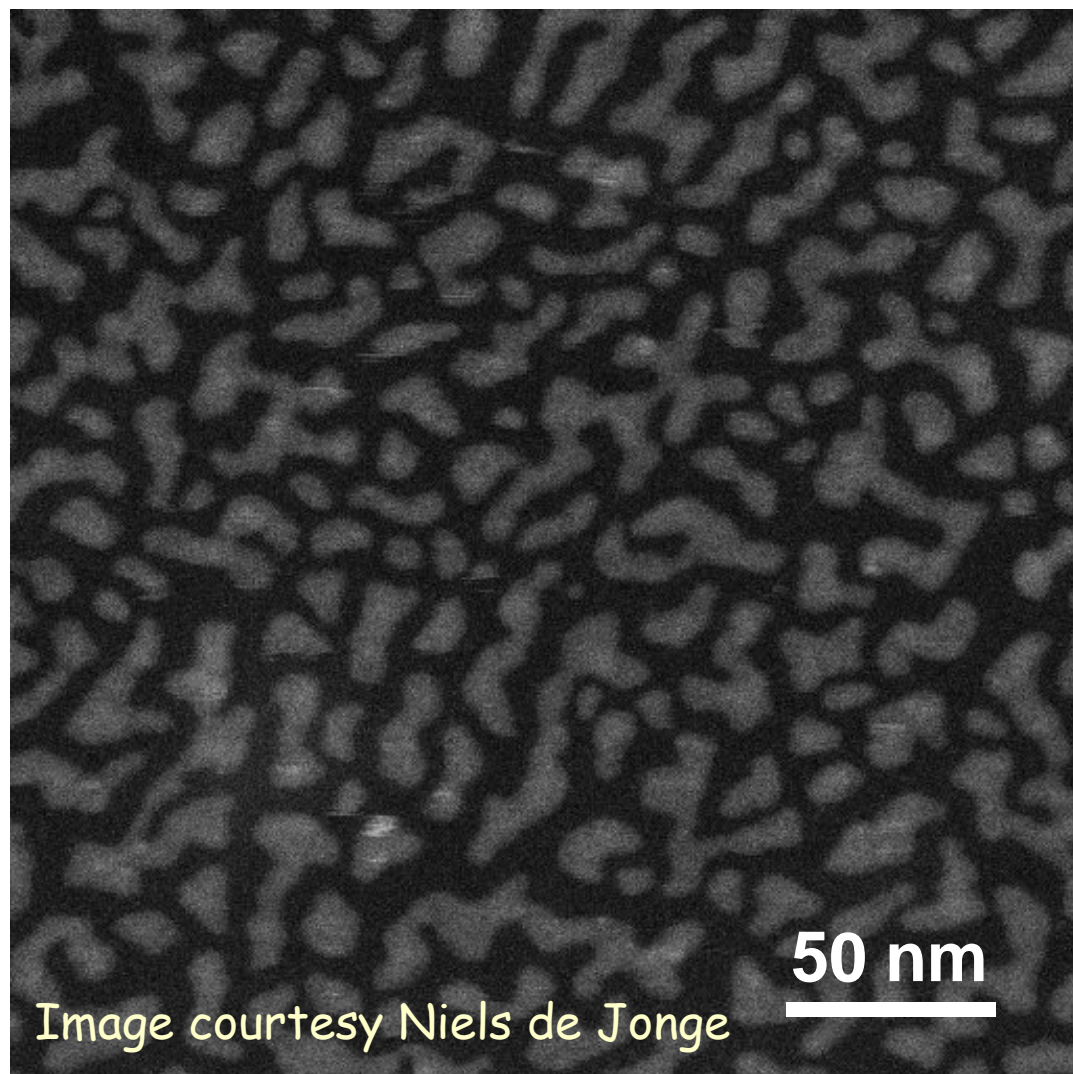
Holder design by Niels de Jonge (HTML/SHaRE)

Current Research Areas

- ✓ Development of methods for high resolution electron imaging in liquids, or high pressure or high temperature gases
- ✓ Through-flow design allows adjustment and monitoring of all parameters in real time
- ✓ Maintains full imaging and micro-analytical performance of STEM



High Resolution Imaging in Water



- ✓ Gold nano-particles imaged in HAADF mode while floating at a depth of 7 microns in water

- ✓ Resolution ~ 2.5 nm

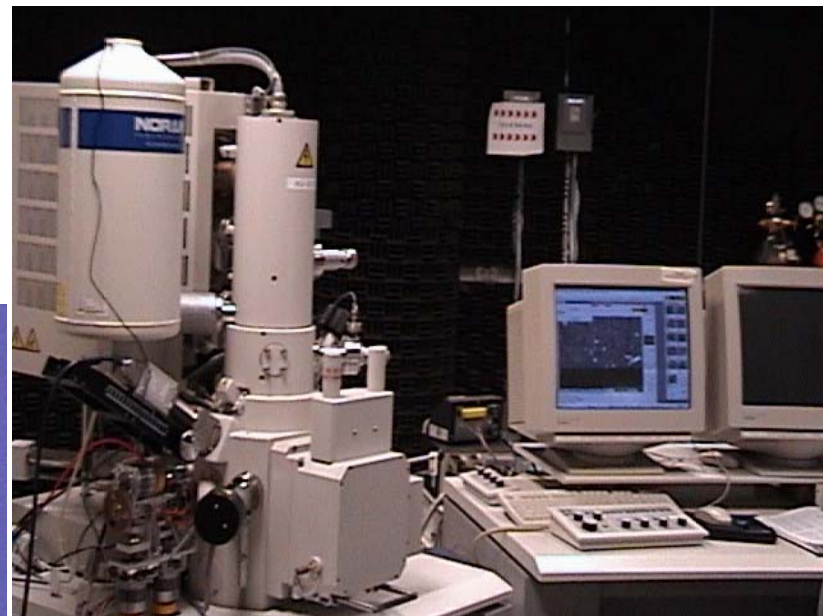
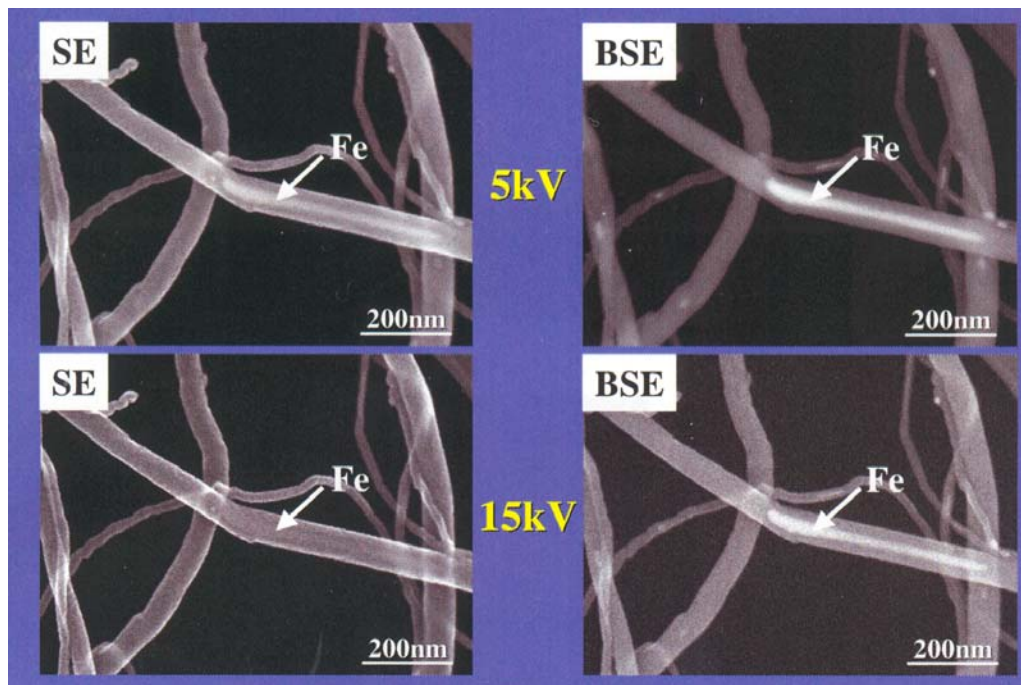
Proposed study areas

- ✓ Catalysis in gaseous or liquid environments
- ✓ Effect of surface nano-topography on electrolysis for hydrogen production
- ✓ Nanotoxicity, nano-particle transport into cells and tissue



CNMS Scanning Electron Microscopes

- ✓ **Hitachi S4700** FEGSEM
- ✓ Dual SE detectors, BSE detector 5-axis motorized stage and EDS system



High resolution imaging for nano-materials science over a wide range of energies and in different signal modes

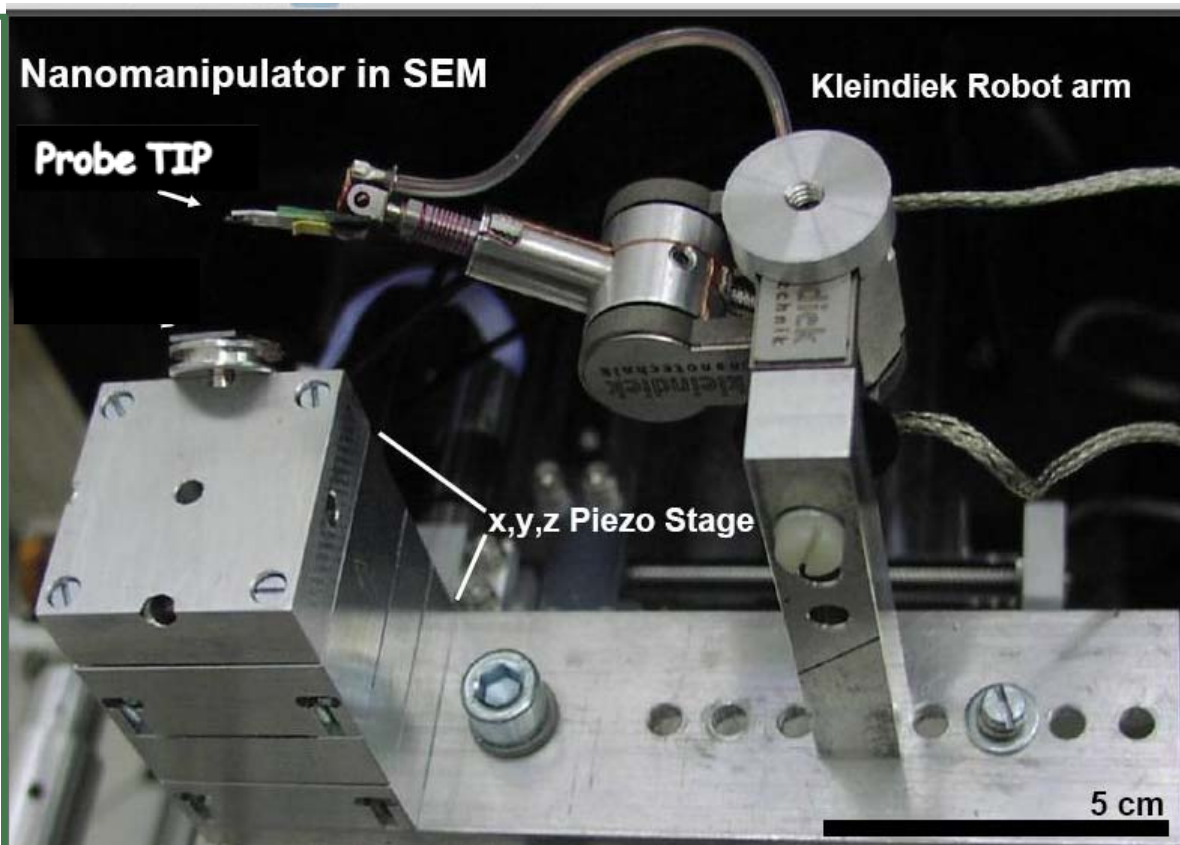


CNMS Goal: Multifunctional Imaging

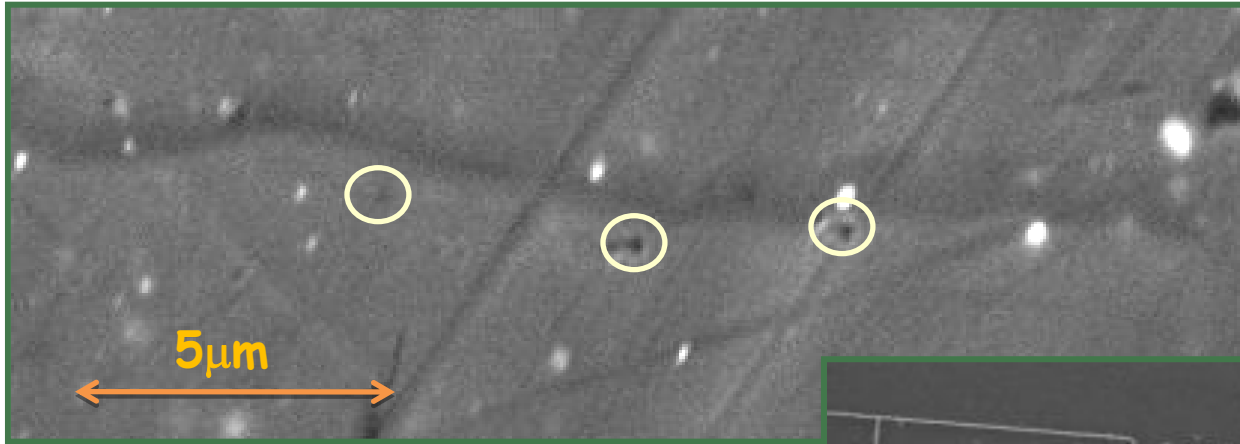
- Hitachi S4700 FEG SEM with Oxford/4Pi EDS system
- Two Kleindiek Nano-manipulators, with force and electrical sensors, installed in sample chamber

Current Research Areas

- ✓ Electrical conductivity imaging (ECI) to trace connectivity in 3-D CNT and graphene mats
- ✓ Nano-indent hardness measurements
- ✓ Electrical and mechanical measurements on single nanowires, ribbons, or tubes
- ✓ Pick-up and mounting of nanoparticles microscopy

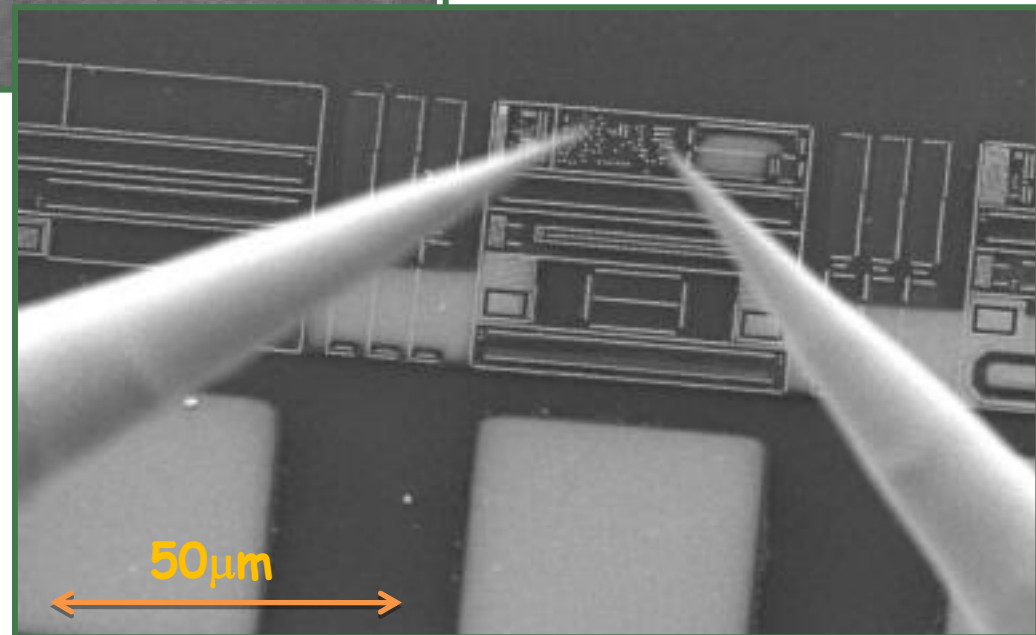


Force and Electrical Probing



Nano-indentations for measurements in deformed copper foil

Tips, connected as test probes, measure resistance of circuit elements in experimental device structure



CNMS NanoFab has an In-Cleanroom SEM for Imaging full-sized Wafers



JEOL JSM-7400 FEG SEM System

- 0.5 to 30 kV accelerating voltage
- 1.0 nm resolution (15 kV SEM mode)
- sample sizes up to 200 mm



Microscopy at CNMS

- Recent software upgrades offer advanced image and analysis modes
- Hitachi SEM and STEM now have identical user interfaces – speeds training and reduces chance of errors and accidents
- Typical usage rate in excess of 80% of available machine time through the year
- Instruments provide ‘uptime’ in excess of 90% of scheduled availability
- New techniques imaging under liquids and new technology such as *in situ* nano-manipulators provide important capabilities for nanoscience

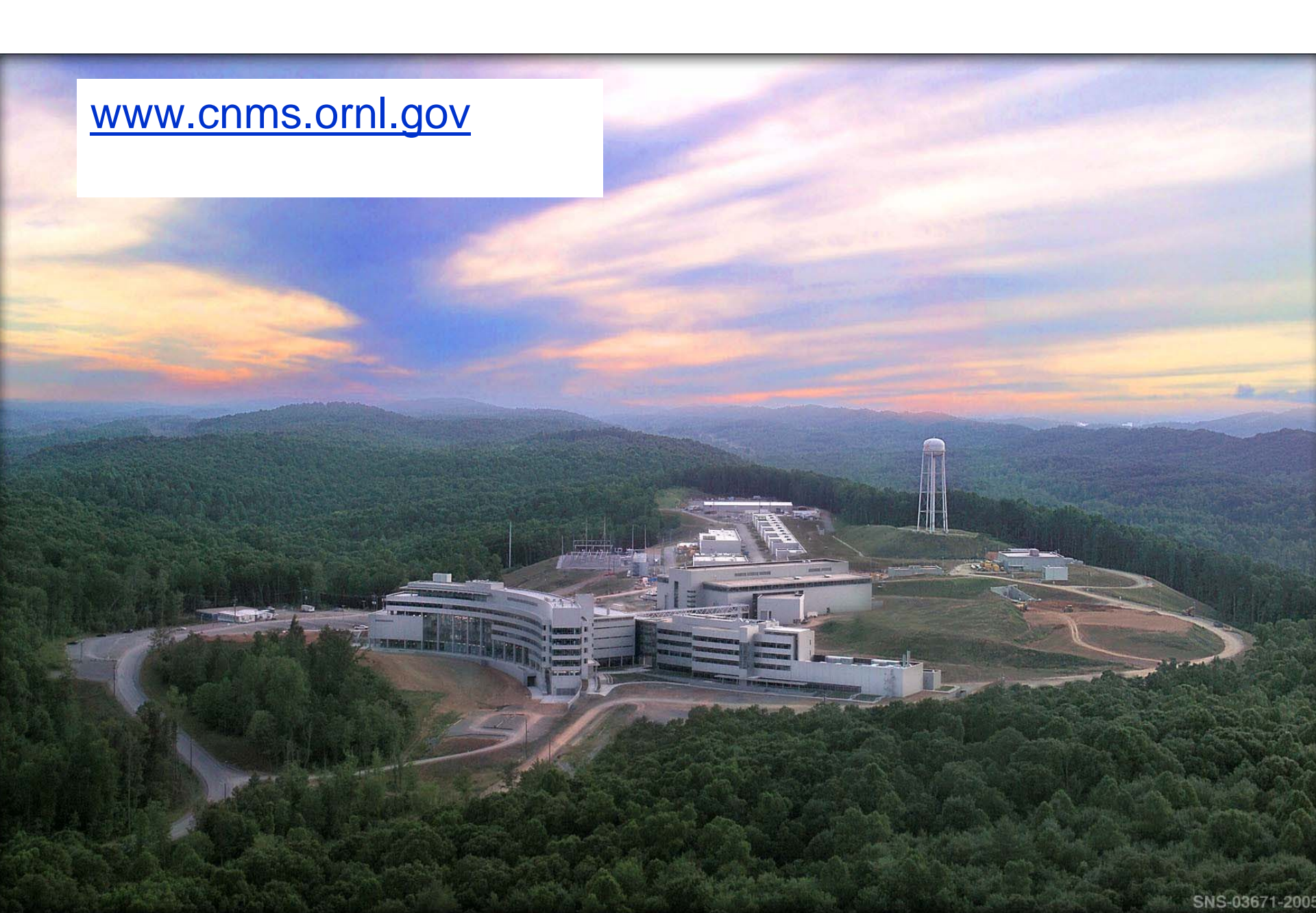


From a broad survey of the CNMS user community: Needs for the future

- **High end S/TEMs and SEMs:**
 - Dedicated high-resolution (aberration corrected) instrument for imaging incorporating electron spectroscopy, hot stage and in situ video, and equipped to interact – mechanically, thermally and electrically – with nanomaterials
 - “Multifunction” quantitative measurements – possible in environments....
 - Environmental TEM and SEM
- **“Everyone” wants access to capabilities offered by a Titan or the equivalent....**



www.cnms.ornl.gov



SNS-03671-2005